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STUDIES ON THE SYNTHESIS OF BORON HYDRIDE SYSTEMS(U)
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STUDIES ON THE SYNTHESSES OF BORON HYDRIDE SYSTEMS

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For the Period
April 1, 1982 - March 31, 1985

Sheldon G. Shore

May 25, 1985

U. S. ARMY RESEARCH OFFICE
Research Triangle Park, North Carolina 27709

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THE OHIO STATE UNIVERSITY

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An improved synthesis of $B_{10}H_{14}$ from B_5H_9 has been developed giving 50 - 58% yields. Application of B_5H_9 as a potential feedstock for preparation of higher boron hydride systems has been demonstrated through the syntheses of a variety of materials in yields, in general, equal to or better than previous methods.		

Among the materials prepared from B_5H_9 are $B_{10}H_{14}$, $n-B_{18}H_{22}$, $B_9H_{13}L$ ($L = S(CH_3)_2$, $S(C_2H_5)_2$, $S(C_6H_5)_2$, $S(t-C_4H_9)_2$, $P(C_6H_5)_3$), nido-5,6-(CH₃)₂-5,6-C₂B₈H₁₀ and nido-C₆H₁₃(H)-5,6-C₂B₈H₁₀).

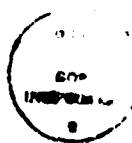
An iodide complex of $B_{10}H_{14}$, $[B_{10}H_{14}I]^-$ has been prepared and studied in detail. It is a remarkably stable species. The X-ray crystal structure of the $[2,4-I_2B_{10}H_{12}I]^-$ has been determined. The structure reveals that the I^- ion interacts with the open end of the B_{10} molecule and effectively rests upon the bridging H atoms.

The structure of the $[B_2H_7]^-$ has been determined by X-ray and neutron diffraction studies. The anion has a bent 3-center hydrogen bridge bond which is of interest since prior theoretical work predicted that the ion would be linear.

A new, simple preparation of B_5H_{11} is described. Reduction of B_5H_9 by alkali metal naphthalides and subsequent protonation by HCl has produced B_5H_{11} in 22% yield.

Our earlier development of a simple, safe, and high yield synthesis of B_4H_{10} has made available potentially convenient routes to metallapentaboranes through the reaction of $[B_4H_9]^-$ with suitable transition metal complexes. These possibilities have been pursued as adjuncts to our boron hydride syntheses. The following metallapentaboranes have been prepared and characterized.

$[Ir(h^4-C_4H_6)(CO)\{P(CH_3)_2C_6H_5\}_2]$, $[Ni(h^3-B_4H_8)\{(CH_2)P(C_6H_5)_2\}_2]$, $[Cu(h^3-B_4H_9)\{P(C_6H_5)_3\}_2]$, and $[Rh(h^3-B_4H_8)H\{P(C_6H_5)_3\}_2]$.



FORWARD

Over the past three years, under ARO support, the principal objective of this program has been to fully develop and to extend ~~our~~ earlier work on the conversion of pentaborane(9), B_5H_9 , to potentially useful higher boron hydride systems. The existence of a large quantity of B_5H_9 in U. S. government storage provided the impetus for this work. On the whole, this objective has been achieved. ^{Achieved were} ~~We have made~~ significant improvements over ^{an} ~~our~~ earlier synthesis of decaborane(14), $B_{10}H_{14}$, ^{it has been} both from the stand point of yield and safety. Furthermore, ~~we have~~ demonstrated that B_5H_9 can be converted to a number of higher boron hydride species through "one-pot" procedures which give yields of products, in general, equivalent to or better than obtained by other methods. Thus, B_5H_9 in storage is a potential feedstock for these materials.

In addition to ^{these} above efforts, during the grant period ~~we~~ ^{were} undertook studies of the general chemistry of boron hydride ^{under taken} and report some interesting new developments, ^{are} reported.

*Additional beyond: carboranes; metallaboranes;
feedstock; tetraborane; Synthesis (chemistry).*

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AN IMPROVED SYNTHESIS OF DECABORANE(14) AND THE
APPLICATION OF
PENTABORANE(9) AS A FEEDSTOCK FOR HIGHER BORON HYDRIDE
SYSTEMS

Our earlier synthesis of $B_{10}H_{14}$ (Technical Report III "Studies on the Conversion of Pentaborane(9) to Decaborane(14)") has been evaluated in a report by Raymond A. Freeman, CPT.EN. USAR to ARO "Production of Decaborane from Pentaborane", August 1983. We were able to respond positively to questions raised in this evaluation. These questions are concerned with the possibility of replacing THF with a solvent of higher flash point and the possibility of extracting $B_{10}H_{14}$ from the reaction mixture rather than subliming it from the mixture. We were able to modify our procedure, giving a significantly improved and safer synthesis. These modifications are described in detail in our Technical Report Part IV "Studies on the Conversion of Pentaborane(9) to Decaborane(14)", March 24, 1984 and in our Progress Report for the period July 1, 1984 - December 31, 1984. Our revised procedure consistently gives yields of 50-58% compared to consistent yields of 42-45% yields which were obtained earlier.

The principal new features of our procedure are: 1. Glyme has a higher flash point than THF and replaces THF in the current procedure. 2. The ratio of reactants B_5H_9/NaH has been reduced from 2.0/1.0 to 1.8/1.0 thereby conserving B_5H_9 . 3. The sublimation procedure used to isolate $B_{10}H_{14}$ has been modified. It was found that high quality $B_{10}H_{14}$ (~50% yield) can be isolated from the reaction mixture if the sublimation is carried out at 40-45°C under vacuum ($<10^3$ mmHg). The more vigorous sublimation technique reported earlier gives higher yields (~60%), but the product is not as pure as that obtained by the milder sublimation procedure. The necessary purification step required to obtain $B_{10}H_{14}$ of equivalent quality to that obtained from the milder sublimation procedure gives a final yield of 50 - 55%.

4. An alternative route to isolating $B_{10}H_{14}$ from the reaction mixture has been developed. In addition to the sublimation route used previously, it was also possible to isolate $B_{10}H_{14}$ in about 50% yield by extracting it in $(n-C_4H_9)_2O$. This procedure is advantageous in that the extract can be used directly in the preparation of n-hexylcarborane.

In the course of developing the preparation of $B_{10}H_{14}$, it became clear to us that other higher boron hydride species, potentially useful materials, could also be produced from B_5H_9 , which is an attractive possibility in view the fact that an appreciable amount of B_5H_9 is currently stockpiled by the U. S. Government. The principal results of this effort are detailed in our Technical Report V "Pentaborane(9) as a Feedstock for the Preparation of Higher Boron Hydride Systems", September 15, 1984. Subsequent to this report we extended our efforts to the preparation of an icosahedral carborane $1,2-(CH_3)_2-1,2-C_2B_{10}H_{10}$ from B_5H_9 as a starting material. The procedure is described in our Progress Report for the period July 1, 1984 - December 31, 1984.

Scheme I indicates the variety of materials we have obtained from B_5H_9 as the starting point. Note that the key factor in all of these syntheses is the initial preparation of $[B_9H_{14}]^{1-}$, generated as an intermediate, from B_5H_9 without the necessity of isolating it. It is of some interest that one of advantages of the syntheses outlined in Scheme I is that $B_{10}H_{14}$ is not required in order to obtain $B_9H_{13}L$ or $n-B_{18}H_{22}$. Earlier procedures from other laboratories first converted $B_{10}H_{14}$ to $[B_9H_{14}]^-$ or other $B_{10}H_{14}$ derivatives which were then converted to $B_9H_{13}L^1$ and $n-B_{18}H_{22}^{1a,1b,2}$. Earlier procedures for obtaining the carborane $R_2C_2B_8H_{10}^3$ involved the rare boron hydride $B_8H_{12}^3$ or degradation of higher carboranes obtained from $B_{10}H_{14}$. The procedures developed, for the most part, are "one-pot" syntheses and the yields of products, in general are equivalent to or exceed yields

of these materials prepared by other methods.

In order to maximize yields of products outlined in Scheme I, the conversion of B_5H_9 to $[B_9H_{14}]^-$ was studied in detail as a function of mole ratio of reactants, reaction time, and temperature using boron-11 NMR spectroscopy to monitor the reaction. The optimum ratio of reactants was found to be 1.8 B_5H_9 /1 NaH. Listed below are yields of products obtained from $[B_9H_{14}]^-$ prepared from reactants in this mole ratio.

Compound	Yield based upon B_5H_9
$B_9H_{13}S(C_2H_5)_2$	80%
$B_9H_{13}S(CH_3)_2$	78%
$B_9H_{13}S(t-C_4H_9)_2$	80%
$B_9H_{13}S(C_6H_5)_2$	80%
$B_9H_{13}P(C_6H_5)_3$	82%
$n-B_{18}H_{22}$	39%
$B_{10}H_{14}$	50 - 58%*
<u>nido</u> -5,6- $(CH_3)_2$ -5,6- $C_2B_8H_{10}$	39%
<u>nido</u> - $C_6H_{13}(H)$ -5,6- $C_2B_8H_{10}$	42%**

*Yield depends upon method of isolation

**Mixture of isomers

AN IODIDE COMPLEX OF $B_{10}H_{14}$, $[B_{10}H_{14}I]^-$

We found that $B_{10}H_{14}$ reacts with iodide salts of complex cations ($[N(\underline{n}-C_4H_9)_4]^+$, $[P(C_6H_5)_3CH_3]^+$, $[PPN]^+$) to form the complex ion $[B_{10}H_{14}I]^-$.

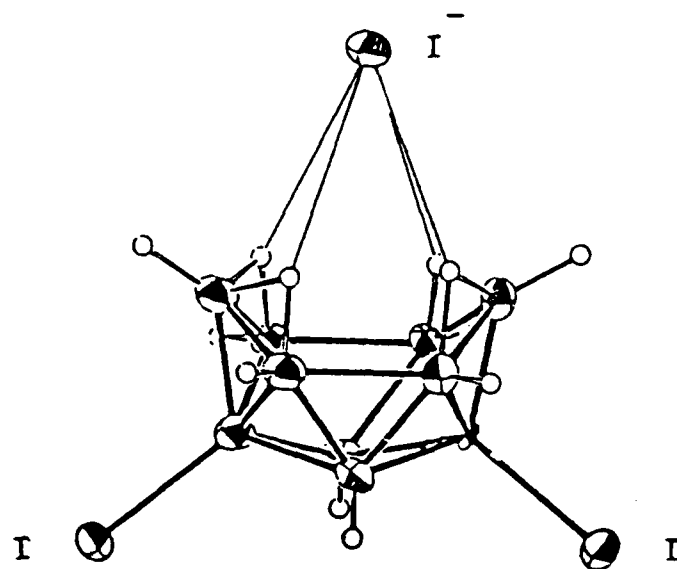


Decaborane(14) reacts with iodide salts of the complex cations shown above upon mixing the reactants, and the addition of THF or CH_2Cl_2 results in the formation of deep yellow solutions. Boron-11 and proton NMR solution spectra show that although the apparent C_{2v} symmetry of the B_{10} framework is preserved, the chemical shifts of the 6,9 boron atoms and the bridging protons are markedly perturbed by the presence of I^- . The solution Raman spectrum of the complex also shows a marked perturbation of the bridge stretching frequencies.

The solid salts of the $[B_{10}H_{14}I]^-$ ion cannot be prepared with alkali metal iodides, but with the complex cations shown above the salts are remarkably stable. Thus, decaborane(14) is volatile at room temperature, but cannot be sublimed away from $[N(\underline{n}-C_4H_9)_4][B_{10}H_{14}I]$ at $95^\circ C$. under vacuum (10^{-3} mm Hg). The formula of the $[B_{10}H_{14}I]^-$ ion in CH_2Cl_2 solution was established by observing the absorption maximum (355 nm) while performing Job's continuous variations experiments. From these experiments the equilibrium constant for the formation of $[N(\underline{n}-C_4H_9)_4][B_{10}H_{14}I]$ is 127 at $268^\circ K$.

While X-ray structures of salts of the $[B_{10}H_{14}I]^-$ ion could not be determined because of disorder, the X-ray structure of $[P(C_6H_5)_3CH_3][2,4-I_2B_{10}H_{12}I]$ was determined and the structure of the $[2,4-I_2B_{10}H_{12}I]^-$ ion is shown below. This structure reveals that the I^- ion interacts with the open-end of the B_{10} molecule and effectively rests upon the bridging H atoms. The solution

spectra, Raman and NMR, are consistent with the solid state structure.



Structure of $[2,4-I_2B_{10}H_{12}I]^-$

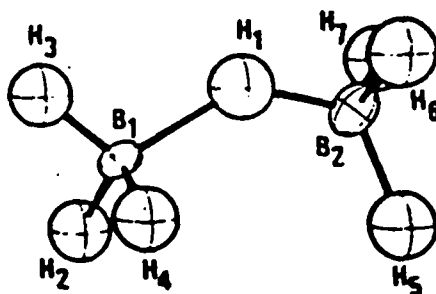
A NEW PREPARATION OF PENTABORANE(11), B_5H_{11}

Earlier work from this laboratory described the preparation of B_5H_{11} ^{4,5} through a hydride abstraction reaction. At the time that synthesis was reported^{4,5}, it was by far the best and most convenient method for obtaining this once rare boron hydride. In the course of studying the reduction of boron hydrides with alkali metal reducing agents in the presence of an electron carrier, we have developed a new and what promises to be a more convenient synthesis for B_5H_{11} than previously available. We find that B_5H_9 is reduced by potassium naphthalide and also by cesium naphthalide in THF to give what appears to be the cesium and potassium salts of the $[B_5H_9]^{2-}$ ion. Treatment of these salts with HCl gives B_5H_{11} . Yields of the order of 22% B_5H_{11} have been obtained to

date. While 60% yields of B_5H_{11} were obtained by our earlier procedure, the present procedure is much simpler and with further study will probably give significantly larger yields.

THE STRUCTURE OF THE $[B_2H_7]^-$ ION

In collaboration with Professor Robert Bau at the University of Southern California the structure of the $[B_2H_7]^-$ ion as the $[PPN]^+$ salt was determined by X-ray⁶ and neutron diffraction⁷ and is shown below. This anion has a bent $(136(4)^\circ)$ 3-center hydrogen bridge bond which is of interest since prior theoretical work⁸ predicted that the ion would be linear. As a result of our study, theoreticians have revised their calculations and now predict⁹ that the structure should be bent.

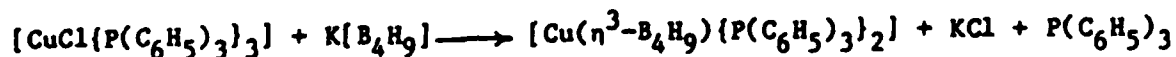
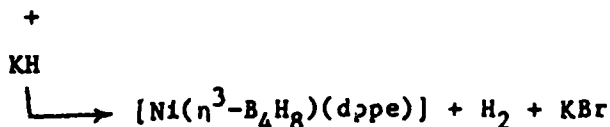
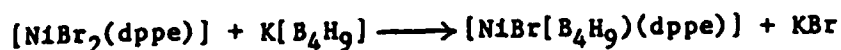
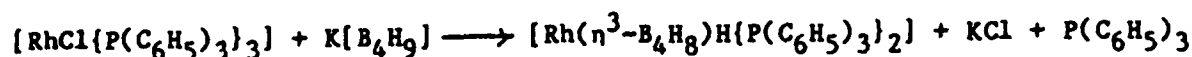
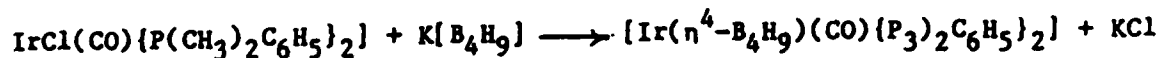


Structure of $[B_2H_7]^-$

METALLA- DERIVATIVES OF TETRABORANE(10)

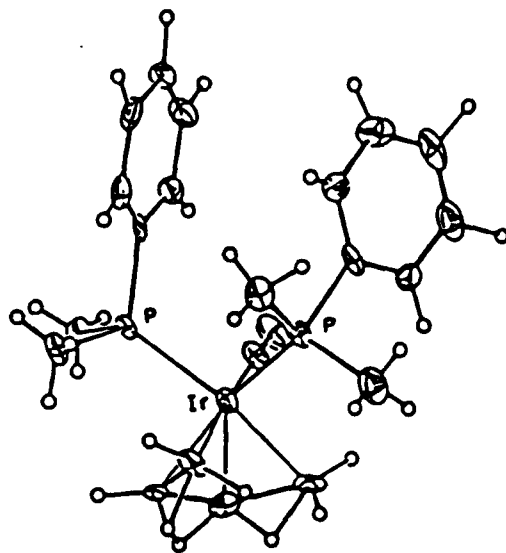
Our development of^a relatively simple, safe, and high yield synthesis of B_4H_{10} has made available potentially convenient routes to metallapentaboranes through the reaction of $[B_4H_9]^-$ with suitable transition metal complexes. We have pursued these possibilities as adjuncts to our boron hydride syntheses.

Although metallapentaboranes have been known for some years¹⁰ they have, in general, been synthesized by indirect methods from either higher boron hydrides or borane anions other than $[B_4H_9]^-$. Furthermore, yields have usually been small. We have prepared in good yields irida, rhoda, nickla-, and cuprapentaboranes¹¹ from the reactions of complexes of the metals with the $[B_4H_9]^-$ ion.



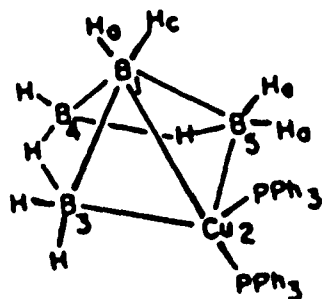
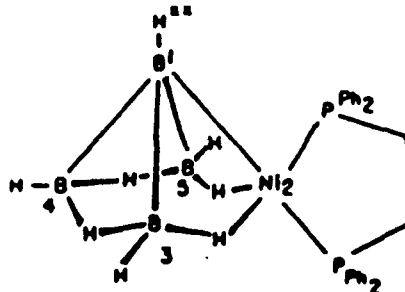
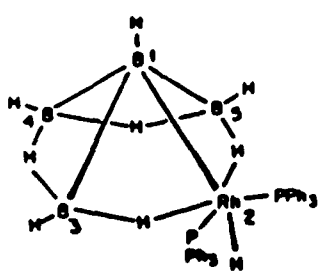
The compounds prepared in the above reactions are unique, not only because they are the first metallapentaboranes prepared from $[B_4H_9]^-$, but also because the metals employed in the study bind to the B_4 ligands in ways which differ somewhat from the bonding exhibited in their complexes with other borane ligands¹⁰.

Each of the metallapentaboranes prepared has a different molecular structure. The structure of $[Ir(h^4-B_4H_9)(CO)\{P(CH_3)_2C_6H_5\}_2]$ was determined by X-ray diffraction¹¹ and is shown below. It is an open four-sided pyramidal arrangement of an arachno- $[IrB_4]$ cluster. It is the structural analogue of B_5H_{11} and it is also isoelectronic and isostructural with the 1,3-butadiene-iridium complex $[Ir(h^4-C_4H_6)(CO)\{P(CH_3)_2C_6H_5\}_2]$.



Structure of $[Ir(h^4-C_4H_6)(CO)\{P(CH_3)_2C_6H_5\}_2]$

The structures of the rhoda, nickla, and cupra-complexes, shown below have been inferred from boron-11 and proton NMR spectra. While each complex is best described as a closed four-sided pyramid, the structures proposed, (shown below) seem to differ in detail. Thus $[\text{Rh}(\text{h}^3\text{-B}_4\text{H}_8)\text{H}\{\text{P}(\text{C}_6\text{H}_5)_3\}_2]$ contains a B_4H_8 unit and a rhodium-hydrogen bond which is indicative of oxidative addition of the $[\text{B}_4\text{H}_9]^-$ ion to the Rh atom in the synthetic reaction shown above. The complex $[\text{Ni}(\text{h}^3\text{-B}_4\text{H}_8)\{(\text{CH}_2)\text{P}(\text{C}_6\text{H}_5)_2\}_2]$ does have a B_4H_8 unit but no terminal nickel-hydrogen bond. Its synthesis depends upon a strategy which involves a deprotonation step after the formation of an initial nickel-borane complex. The complex $[\text{Cu}(\text{h}^3\text{-B}_4\text{H}_8)\{(\text{CH}_2)\text{P}(\text{C}_6\text{H}_5)_2\}_2]$ appears to have two terminal hydrogens on the apical boron atom, an arrangement which is uncommon.



Proposed structures of $[\text{Rh}(\text{h}^3\text{-B}_4\text{H}_8)\text{H}\{\text{P}(\text{C}_6\text{H}_5)_3\}_2]$, $[\text{Ni}(\text{h}^3\text{-B}_4\text{H}_8)\{(\text{CH}_2)\text{P}(\text{C}_6\text{H}_5)_2\}_2]$, and $[\text{Cu}(\text{h}^3\text{-B}_4\text{H}_8)\{(\text{CH}_2)\text{P}(\text{C}_6\text{H}_5)_2\}_2]$

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SCIENTIFIC PERSONEL

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